

## Determination of antioxidants (Irganox 1076 and Irganox 1010) in polyethylene using thermal desorption and reactive pyrolysis – Part 1

**[Background]** Plastic products often contain a number of additives which give the products desirable physical and chemical properties. Irganox 1076 and 1010 are sterically hindered phenolic antioxidants and are widely used in the formulations of plastics, lubricants, adhesives and coatings. In this report, GC/MS determination of Irganox 1076 (MW 530) and Irganox 1010 (MW 1178) is described. Irganox 1076 in polyethylene (PE) is determined using thermal desorption (TD)-GC/MS. Irganox 1010 has a high boiling point and consequently, TD cannot be used. Irganox 1010 in PE can be hydrolyzed and methylated using tetramethyl ammonium hydroxide (TMAH). The methyl derivative has a much lower boiling point and it can be easily vaporized and determined using GC/MS.

**[Experimental]** PE-pellets, spiked with either Irganox 1076 (340 ppm) or Irganox 1010 (470 ppm), were pulverized. The spiked PE samples containing Irganox 1076 and Irganox 1010 were respectively analyzed by TD-GC/MS and reactive pyrolysis (RxPy)-GC/MS. In RxPy-GC/MS, TMAH was added to the sample cup containing the spiked PE prior to analysis. Standard addition method was used to generate calibration curves. The analysis was done using a Multi-Shot Pyrolyzer (EGA/PY-3030D, Frontier Labs) which was directly interfaced to the split injector of a GC/MS system.

**[Results]** TD chromatogram of PE containing Irganox 1076 is shown in Fig. 1a. The large peak at 26.8 min is assigned as Irganox 1076. Figure 1b shows the total ion chromatogram of PE containing Irganox 1010 treated by hydrolysis and methylation using TMAH. The large peak at 17.9 min is assigned as the methyl derivative of Irganox 1010. Using the standard addition method, the concentrations of Irganox 1076 and Irganox 1010 were respectively determined to be 374 ppm (10% error) and 429 ppm (9% error).

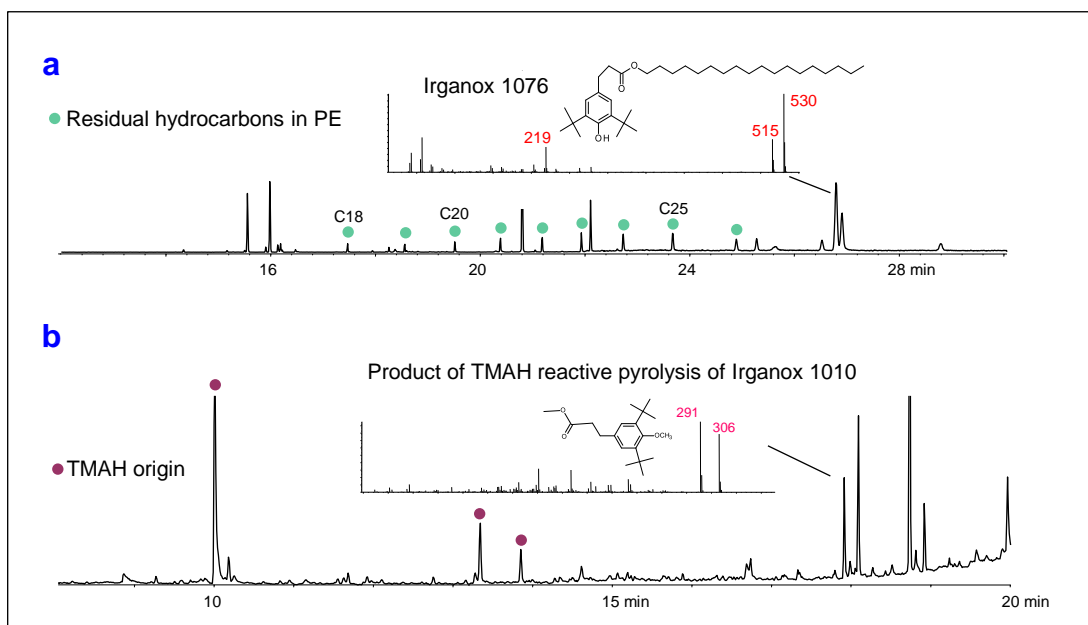


Fig. 1 a: Chromatogram of PE with Irganox 1076 obtained by thermal desorption  
 b: Pyrogram of PE with Irganox 1010 obtained by reactive pyrolysis

Furnace temp.: 260°C (reactive pyrolysis), 320°C (thermal desorption)  
 GC oven: 40-150°C (10 °C/min) - 320°C (20 °C/min, 3 min hold)  
 Separation column: Ultra ALLOY+5 (5% diphenyl 95% dimethylsiloxane), L=30 m, i.d.=0.25 mm, df=0.25 µm  
 Split ratio: 1/30, Sample wt.: approx. 100 µg, 25 wt% TMAH methanol solution: 20 µL

**Keywords :** Irganox 1010, Irganox 1076, Thermal desorption, Reactive pyrolysis, TMAH

**Products used :** Multi-functional pyrolyzer, UA+-5

**Applications :** Analysis of additives in polymer

**Related technical notes :** PYA1-081E

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